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(54) A moisture sensor formed by an electrical capacitor

(57) A moisture sensor formed by an electrical capacitor has as one electrode a silicon crystal 1, as its second electrode a water transmissive gold layer 5, and as dielectric a layer 3 of moisture sensitive silicon dioxide with impurity ions (Ca, Mg, Al). An additional layer 2 of silicon dioxide and/or silicon nitride can be provided. The sensor can be mounted together with an evaluation circuit on a single chip. The moisture sensitive layer may be produced by ion-implantation, or by spinning from a solution containing, e.g. orthosilicic ethyl ester, methanol, aqueous calcium chloride and nitric acid at a pH of 1. It was noted that even the layer 2 of silicon dioxide produced by oxidation of the silicon substrate showed moisture sensitivity.

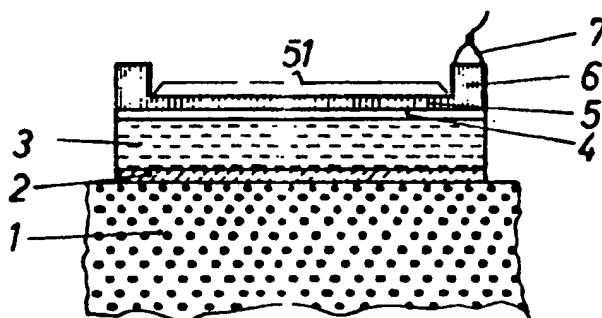


FIG. 1

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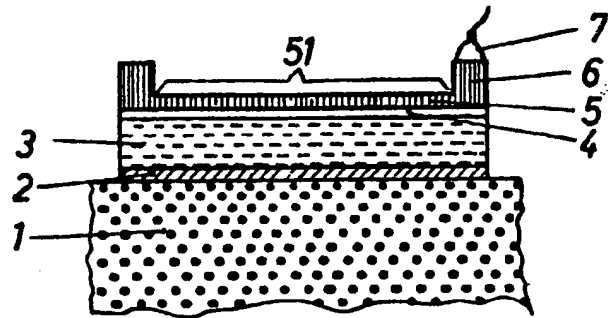


FIG. 1

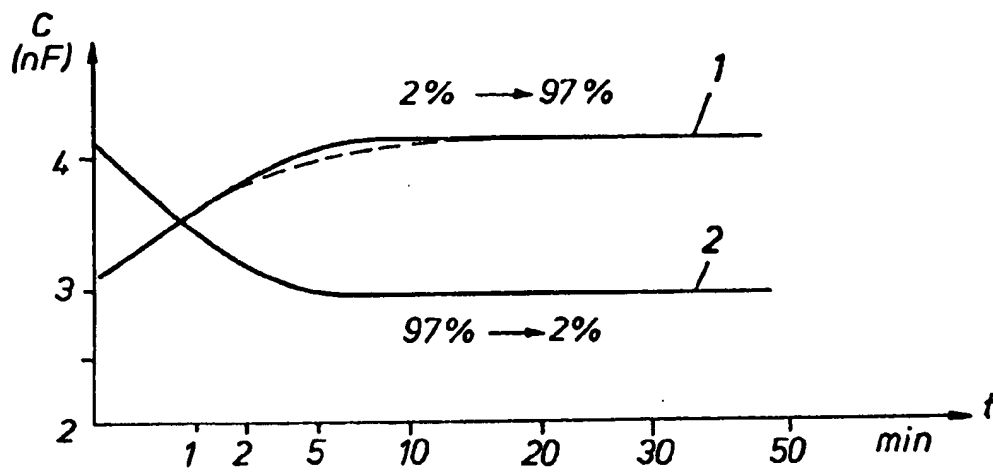


FIG. 2

## SPECIFICATION

## A moisture sensor formed by an electrical capacitor

5 The invention concerns a moisture sensor formed by an electrical capacitor, for measurement of the relative humidity of the air, and having a metal substrate as one electrode, a water transmissive metallic layer as the other electrode, and a moisture sensitive layer mainly forming the dielectric, and also a method for production of the sensor.

10 The development of integrated circuits and micro-processors makes available many new possibilities for the handling of signals. The use of integration techniques for the measurement and control of the physical processes is, however, only possible with advantage if acceptable signal generators with a sufficiently great electrical output are available.

15 For hygrometric measurements in air, hair hygrometers have been almost exclusively used up to now. This does not however satisfy the requirement for an electrical output. Therefore, moisture sensors have been developed which are formed by a capacitor of which the dielectric is moisture sensitive. A large proportion of the commercially available moisture sensors contain an organic polymer as the moisture sensitive layer. These polymers have, however, a limited life, and a limited range for variation of the capacitance upon changes in the ambient humidity. Moisture sensors with aluminium oxide as the sensitive layer seem better suited for this job, special production methods being necessary to ensure that the layer responds to the ambient water content.

20 These sensors are individual elements, and as a rule therefore their output data is not suitable for use with micro processors. It is desirable not only to have electrical matching between the sensor and the circuit operating on the signal, but also integration of both components on one silicon chip. At present due to the large differences between integrated circuit technology and that for the production of the sensor, such a combination is not possible.

25 The invention is therefore based upon the problem of improving moisture sensors formed by an electrical capacitor, of the type mentioned in the introduction, beyond the state of the art. In particular, it should be possible without problems to integrate it with the evaluation circuit, it should have sufficient sensitivity, should supply measured values of reverse sign in the shortest possible response time, and should be easy to manufacture.

30 According to a first facet of the invention, there is provided a moisture sensor, for measurement of the moisture content of gases, formed by an electrical capacitor means having two electrodes and a dielectric layer between, wherein a silicon monocrystal substrate forms one electrode, a moisture sensitive layer of silicon dioxide forms the dielectric layer, and a water-transmissive metallic layer forms the other electrode. If impurity ions from elements of the first to the third main groups of the periodic table are included in the moisture sensitive silicon dioxide layer, one achieves a moisture sensor of very much higher sensitivity. With an additional

silicon dioxide layer and/or a silicon nitride layer between the silicon substrate and the moisture sensitive layer, the electrical impedance between the electrodes of the capacitor is increased in an advantageous manner.

70 According to a further feature of the invention, there is provided a method for the production of a moisture sensor having a silicon dioxide layer with impurity ions as a moisture sensitive layer, wherein said layer is produced by a spin-on process from a solution, and the ions are already present in the solution. By production of the moisture sensitive layer using a spin-on process from a solution, the impurity ions are very easily introduced into the layer by introduction of the ions into the solution. Exact control of the number of impurity ions in the silicon dioxide layer is possible with the help of ion implantation.

80 In order that the invention shall be clearly understood, various exemplary embodiments thereof will now be described with reference to the drawings, in which:

Figure 1 shows a cross-section through a moisture sensor according to the invention;

90 Figure 2 shows a graph of the change in capacitance with change in relative air humidity from 2 to 97% in dependence on time.

The major feature of the invention lies in that a silicon dioxide layer is used as the moisture sensitive dielectric. This is preferably doped additionally with ions of the I to III main group of the periodic table, for example Ca, Mg, Al or from the sub-groups, for example Cu. Silicon dioxide is known as a drying agent in the form of silicagel. The basis of the invention is the recognition that by including ions in an amorphous silicon dioxide layer the hygroscopic characteristics of this substance can be so altered that the absorption and excretion of water is reversible, and occurs sufficiently quickly that within a short time an equilibrium state with the moisture content of the ambient air is reached. The sensitivity, response time and measuring range are variable within wide limits according to the kind and amount of the ions introduced.

100 Semi-conductor technology knows many processes for the production of silicon dioxide layers. In order simply to obtain the ion doping necessary according to the invention, deposition from a solution is used. This method is known from DE-OS 24 47 204. Alternatively, the ions can be introduced by ion implantation. This process has long been used and approved in semi-conductor technology.

110 In addition, if the moisture sensitive layer is deposited on a silicon monocrystal, all the requirements necessary for integration of the sensor and the evaluation circuit on a chip are met.

120 The production of such a sensor will now be described with reference to Figure 1. A silicon chip is covered by oxidation in a stream of oxygen gas with an approximately 50nm thick layer of silicon dioxide. This layer is intended to prevent insulation losses. On to this layer the moisture sensitive layer 3 is deposited using the spin-on process. This layer is produced for example from a solution having the following constitution:

- 100 ml ortho silicic ethylester
- 100 ml methanol
- 100 ml hydrous solution of 1 mol/l  $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ ,
- 5 set to pH = 1 by the addition of  $\text{HNO}_3$

Spinning produces from this solution an approximately 250nm thick silica glass layer 3. This is followed by vapour deposition of a thin chromium layer 4 as an adhesive for the gold layer 5 produced in the same working step. This sequence of layer must be kept so thin that they are still water transmissive. A thicker gold layer 6 (~0.2  $\mu\text{m}$ ) is provided outside the actual sensor surface as an electric contact. This must be produced in a further deposition step after the sensor surface 51 has been covered with photolacquer. The gold layer deposited can then be removed by "lift-off" so that only the ring-shaped contact area is thickened around the sensor surface. Contacts are provided to the silicon substrate 1 and the thickened gold layer 7. Results achieved with such element are shown in Figure 2. This shows the change in capacitance C with change in the relative humidity of the atmosphere from 2% to 97% (curve 1) and back again (Curve 2) in dependence on time t, time on the scale being squared. The change of humidity occurred in a few seconds.

The Figure shows that at first no saturation occurs. Surprisingly it was found that even thermic  $\text{SiO}_2$  (namely the oxide produced by oxidation of silicon in a stream of oxygen at high temperature) responds to moisture. The dashed straight line curves shown in Figure 2 are due to this effect. In an advantageous embodiment of the invention a water resistant layer, preferably  $\text{Si}_3\text{N}_4$  is therefore used in place of the thermally achieved  $\text{SiO}_2$  layer, or in addition thereto (solid line curve).

For integration in a circuit, the capacitance value of the sensor must be matched to the input of the circuit. Normally, suitable capacitance values for this lie in the range from 10 to 100 pF. This can be achieved easily by choice of the sensor surface. The values given in Figure 2 refer to, for example, an active surface of  $5 \times 5 \text{ mm}^2$ . Moreover, for integration, the process steps for producing the sensor and for producing the integrated circuit must be matched. In particular, it must be noted that the moisture sensitive sensor layer cannot be subjected to a high temperature process since it loses its sensitivity at temperatures above  $500^\circ\text{C}$ . On the other hand, temperature processes at lower temperature for specific purposes can be used, in order to set the sensitivity.

In the above-mentioned lift-off process, the already produced gold-layer 5 (Figure 1) received a photolacquer layer which is thicker than the gold layer 6 to be deposited later. After exposure and development of the photolacquer layer the further gold layer 6 is vapour deposited to the desired thickness. When dissolving the photolacquer layer, the solvent attacks the edges of the photolacquer which remain uncovered in the deposition process, and thus removes both the lacquer and with it the gold deposited on the lacquer.

The present invention lays open not only the route to new moisture sensitive materials, but also provides a production method which is compatible with semiconductor technology and which is in addition both simple and thus economic.

## CLAIMS

1. A moisture sensor, for measurement of the moisture content of gases, formed by an electrical capacitor, having a metal substrate as one electrode, a water transmissive metallic layer as the other electrode, and a moisture sensitive layer mainly forming the dielectric, wherein a silicon monocrystal forms the metal substrate, and the moisture sensitive layer consists of silicon dioxide.
2. A sensor as defined in claim 1, wherein impurity ions are introduced into the silicon dioxide.
3. A sensor as defined in claim 2, wherein the impurity ions introduced are ions of the elements of the first to the third main groups or sub-groups of the periodic table.
4. A sensor as defined in claim 2 or 3, wherein an additional layer of silicon dioxide and/or of silicon nitride is provided.
5. A sensor as defined in any preceding claim, which is mounted together with an integrated circuit on a silicon chip.
6. A method for the production of a moisture sensor having a silicon dioxide layer with impurity ions as a moisture sensitive layer, wherein said layer is produced by a spin-on process from a solution, and the ions are already present in the solution.
7. A method as defined in claim 6, wherein impurity ions are also introduced into the silicon dioxide by ion implantation.
8. A method as defined in claim 6 or 7, wherein the sensitivity of the sensor is set to a specific value by a tempering process at high temperature.
9. A moisture sensor substantially as herein described with reference to the accompanying drawings.
10. A method for production of a moisture sensor substantially as herein described with reference to the accompanying drawings.

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